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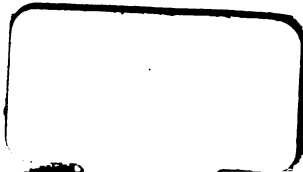
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*Standard Methods of Testing  
and  
Specifications for Cement*





Standard  
Methods of Testing  
and  
Specifications for Cement

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EDITED BY THE SECRETARY

Under the direction of Committee C on Standard Specifications for Cement  
of the American Society for Testing Materials

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*Philadelphia*

PUBLISHED BY THE COMMITTEE

1905



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## INTRODUCTION.

The object of this pamphlet is to assemble under one cover the recent reports of the committees on,

1. Uniform Tests of Cement.
2. Standard Specifications for Cement, and
3. Uniformity in Methods of Analysis for Limestones, Raw Materials and Portland Cements.

These remarks, concerning the steps which led to the preparation and adoption of these reports, are appended as a matter of record.

With the rapid development of the Cement Industry the attainment of a standard of specifications has been most earnestly sought.

The report of the Committee of January 7, 1885, of the American Society of Civil Engineers was the first step in this country in this direction; the report, however, within a very few years became too elastic and indefinite to serve the purpose.

In 1896, at the suggestion of Mr. Richard L. Humphrey, a series of editorials appeared in the *Engineering Record* calling attention to the inadequacy of the 1885 rules and urging the appointment of a new committee to revise them.

Following this a resolution was presented by Mr. Edward P. North at a meeting of the American Society of Civil Engineers, held November 4, 1896, requesting the Board of Direction to report on the advisability of appointing a committee to report on "The Proper Manipulation of the Tests of Cement."

The Board reported at the annual meeting of the society on January 20, 1897, and was instructed to issue a letter ballot. This vote was canvassed, and on July 1, 1897, the board appointed the committee consisting of Messrs. George F. Swain, Alfred Noble, George S. Webster, W. B. W. Howe, L. C. Sabin, O. M. Carter and H. M. York; the latter two members subsequently resigned.

At the annual meeting held on January 16, 1901, the Board of Direction was authorized to increase the membership of this committee to nine, and in accordance therewith appointed Messrs. Spencer B. Newberry, Clifford Richardson, Richard L. Humphrey and F. H. Lewis.

This Committee organized by the election of Prof. George F. Swain Chairman and Mr. Richard L. Humphrey Secretary. On the retirement of Prof. Swain from the chairmanship, under the pressure of personal affairs, Mr. George S. Webster was elected in his place.

The Committee presented to the Society a Progress Report on Uniform Tests of Cement on January 21, 1903.

Efforts were made in 1901 by the American members of the various committees on cement of the International Association for Testing Materials to secure international co-operation in the adoption of a standard specification for cement. This effort came to naught because there were no uniform methods for testing cement and because also of the inability to agree on such methods.

On June 6, 1901, the Board of U. S. Engineers, appointed by the authority of the Secretary of War, presented a report on Testing Hydraulic Cements, and to which were appended Standard Specifications for both Natural and Portland Cements.

At the annual meeting of the American Society for Testing Materials, held on June 15, 1902, the Executive Committee was authorized to appoint a Committee to report on Standard Specifications for Cement, and in accordance therewith named Messrs. Robert W. Lesley, Booth Garrett & Blair, A. W. Dow, Edward M. Hagar, Richard L. Humphrey, Lathbury & Spackman, Andreas Lundteigen, Charles F. McKenna, W. W. Maclay, Spencer B. Newberry, J. M. Porter, Clifford Richardson and George F. Swain, with power to increase its membership subject to the approval of the Executive Committee.

The Committee assembled at the call of Mr. Robert W. Lesley, Member of the Executive Committee and Temporary Chairman, on October 31, 1902, and organized by the election of Prof. George F. Swain, Chairman, Mr. George S. Webster, Vice-Chairman, and Mr. Richard L. Humphrey, Secretary.

The Committee was increased by the addition of the following members: Messrs. T. J. Brady, C. W. Boynton, Spencer Cosby, L. Henry Dumary, A. F. Gerstell, William H. Harding, F. H. Lewis, John B. Lober, Charles A. Matcham, Alfred Noble, H. W. Parkhurst, Joseph T. Richards, L. C. Sabin, H. J. Seaman, S. S. Voorhees, W. J. Wilgus, George S. Webster, H. G. Kelly, Vice-President American Railway Engineering and Maintenance of Way Association, and W. S. Eames, President American Institute of Architects.

Messrs. F. H. Bainbridge, Alfred Noble, H. W. Parkhurst and W. J. Wilgus subsequently resigned from the Committee.

The Committee postponed further action pending the report of the Committee on Uniform Tests of Cement of the American Society of Civil Engineers.

On February 4, 1903, the Committee adopted as a basis for its work the report of the Committee on Uniform Tests of Cement.

In order to obtain data to aid in drafting a Standard Specifications and to test the value of the methods recommended, the Committee sent four samples of natural and five samples of Portland cement

to some thirty prominent laboratories engaged in testing cement with the request that they be tested in accordance with these methods.

The results of these tests were collated, and in accordance with the instructions of the Committee a tentative specification was prepared by the Secretary, Mr. Richard L. Humphrey. These were considered on December 3, 1903, amended and approved March 29, 1904, and adopted by the Committee by letter ballot June 11, 1904; the specifications were approved by the American Society for Testing Materials at its annual meeting June 17, 1904, and adopted by letter ballot of the Society November 14, 1904.

On December 9, 1902, the Association of American Portland Cement Manufacturers appointed a Committee on Standard Specifications for Cement, consisting of Messrs. W. W. Maclay, A. F. Gerstell, D. Millen, B. S. Dunn and T. M. Righter. This Committee was succeeded on January 12, 1904, by the appointment of Messrs. W. W. Maclay, Chairman; A. F. Gerstell, W. H. Harding, S. B. Newberry, Charles A. Matcham, H. J. Seaman and Charles F. Wade. The latter Committee recommended these specifications for, and they were adopted by this Association on June 16, 1904.

The Committee on Masonry of the American Railway Engineering and Maintenance Way Association presented a specification for Portland and natural cement, which was adopted by the Association at the Annual Convention held March 19, 1903, as a recommended provisional standard specification. At the Convention held in March, 1904, no further action was taken in view of the Association having been requested to send a representative to serve on the Committee on Standard Specifications for Cement. Upon the recommendation of the Committee on Masonry, the American Railway Engineering and Maintenance Way Association, in place of the provisional specification, adopted these specifications on March 21, 1905.

These reports on Uniform Tests of Cement and Standard Specifications for Cement are the result of over six years' labor of a thoroughly representative body of experts, covering every field from the manufacturer to the consumer; they stand for the very best thought on the subject.

The various Committees are still in existence and will from time to time recommend such changes as are found by experience to be desirable, thus gradually perfecting the specifications as a whole.

In the meanwhile by the adoption of these specifications a standard of excellence is set which will enable the manufacturer to concentrate his efforts in operating his plant so as to produce uniformly the grade of cement required and at a minimum cost to the consumer.

*Philadelphia, April 14, 1905.*

# American Society of Civil Engineers

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## *Committee on Uniform Tests of Cement*

---

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**W. B. W. HOWE,**  
Consulting Engineer, Spartansburg, S. C.



## *Progress Report of Committee*

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PRESENTED AT THE ANNUAL MEETING, JANUARY 21, 1903, AND  
AMENDED AT THE ANNUAL MEETING, JANUARY 20, 1904.

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Your Committee on Uniform Tests of Cement has devoted much time, and given very careful consideration, to the subject. Frequent meetings have been held, and a number of investigations carried on, some of which cannot be finally reported at this time.

On several matters which have been considered, such as the substitution of a natural sand for the standard quartz, and the tests for the normal consistency and constancy of volume, the Committee has not reached final conclusions.

While not prepared to submit a final report, the Committee feels that it should present a report of progress in order that the Society may be informed of the results of its investigations and conclusions.

The work of the Committee has been confined entirely to the methods for making tests, without attempting to specify what tests should be made.

In order to do full justice to the subject under consideration, it will be necessary to compile and report the results of the experiments now under way, and your Committee asks, therefore, that it be continued.

### **SAMPLING.**

1.—SELECTION OF SAMPLE.—The selection of the sample for testing is a detail that must be left to the discretion of the engineer; the number and the quantity to be taken from each package will depend largely on the importance of the work, the number of tests to be made and the facilities for making them.

2.—The sample shall be a fair average of the contents of the package; it is recommended that, where conditions permit, one barrel in every ten be sampled.

3.—All samples should be passed through a sieve having twenty meshes per linear inch, in order to break up lumps and remove foreign material; this is also a very effective method for mixing them together in order to obtain an average. For determining the characteristics of

a shipment of cement, the individual samples may be mixed and the average tested; where time will permit, however, it is recommended that they be tested separately.

4.—METHOD OF SAMPLING.—Cement in barrels should be sampled through a hole made in the center of one of the staves, midway between the heads, or in the head, by means of an auger or a sampling iron similar to that used by sugar inspectors. If in bags, it should be taken from surface to center.

#### CHEMICAL ANALYSIS.

5.—SIGNIFICANCE.—Chemical analysis may render valuable service in the detection of adulteration of cement with considerable amounts of inert material, such as slag or ground limestone. It is of use, also, in determining whether certain constituents, believed to be harmful when in excess of a certain percentage, as magnesia and sulphuric anhydride, are present in inadmissible proportions. While not recommending a definite limit for these impurities, the Committee would suggest that the most recent and reliable evidence appears to indicate that, for Portland cement, magnesia to the amount of 5 per cent., and sulphuric anhydride to the amount of 1.75 per cent., may safely be considered harmless.

6.—The determination of the principal constituents of cement—silica, alumina, iron oxide and lime—is not conclusive as an indication of quality. Faulty character of cement results more frequently from imperfect preparation of the raw material or defective burning than from incorrect proportions of the constituents. Cement made from very finely-ground material, and thoroughly burned, may contain much more lime than the amount usually present and still be perfectly sound. On the other hand, cements low in lime may, on account of careless preparation of the raw material, be of dangerous character. Further, the ash of the fuel used in burning may so greatly modify the composition of the product as largely to destroy the significance of the results of analysis.

7.—METHOD.—As a method to be followed for the analysis of cement, that proposed by the Committee on Uniformity in the Analysis of Materials for the Portland Cement Industry, of the New York Section of the Society for Chemical Industry, and published in the *Journal* of the Society for January 15, 1902, is recommended.

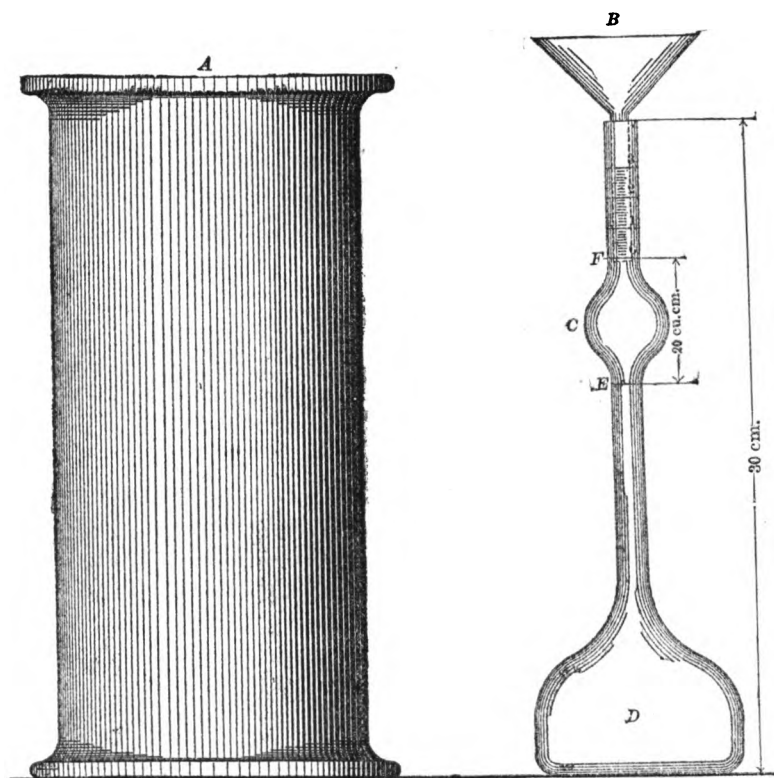
#### SPECIFIC GRAVITY.

8.—SIGNIFICANCE.—The specific gravity of cement is lowered by underburning, adulteration and hydration, but the adulteration must be in considerable quantity to affect the results appreciably.

9.—Inasmuch as the differences in specific gravity are usually very small, great care must be exercised in making the determination.

10.—When properly made, this test affords a quick check for underburning or adulteration.

11.—APPARATUS AND METHOD.—The determination of specific gravity is most conveniently made with Le Chatelier's apparatus. This



LE CHATELIER'S SPECIFIC GRAVITY APPARATUS.

FIG. 1.

consists of a flask (*D*), Fig. 1, of 120 cu. cm. (7.32 cu. ins.) capacity, the neck of which is about 20 cm. (7.87 ins.) long; in the middle of this neck is a bulb (*C*), above and below which are two marks (*F*) and (*E*); the volume between these marks is 20 cu. cm. (1.22 cu. ins.). The neck has a diameter of about 9 mm. (0.35 in.), and is graduated into tenths of cubic centimeters above the mark (*F*).

12.—Benzine (62 degrees Baumé naphtha), or kerosene free from water, should be used in making the determination.

13.—The specific gravity can be determined in two ways:

(1) The flask is filled with either of these liquids to the lower mark (*E*), and 64 gr. (2.25 oz.) of powder, previously dried at 100° Cent. (212° Fahr.) and cooled to the temperature of the liquid, is gradually introduced through the funnel (*B*) [the stem of which extends into the flask to the top of the bulb (*C*)], until the upper mark (*F*) is reached. The difference in weight between the cement remaining and the original quantity (64 gr.) is the weight which has displaced 20 cu. cm.

14.—(2) The whole quantity of the powder is introduced, and the level of the liquid rises to some division of the graduated neck. This reading plus 20 cu. cm. is the volume displaced by 64 gr. of the powder.

15.—The specific gravity is then obtained from the formula:

$$\text{Specific Gravity} = \frac{\text{Weight of Cement}}{\text{Displaced Volume}}$$

16.—The flask, during the operation, is kept immersed in water in a jar (*A*), in order to avoid variations in the temperature of the liquid. The results should agree within 0.01.

17.—A convenient method for cleaning the apparatus is as follows: The flask is inverted over a large vessel, preferably a glass jar, and shaken vertically until the liquid starts to flow freely; it is then held still in a vertical position until empty; the remaining traces of cement can be removed in a similar manner by pouring into the flask a small quantity of clean liquid and repeating the operation.

18.—More accurate determinations may be made with the pycnometer.

#### FINENESS.

19.—SIGNIFICANCE.—It is generally accepted that the coarser particles in cement are practically inert, and it is only the extremely fine powder that possesses adhesive or cementing qualities. The more finely cement is pulverized, all other conditions being the same, the more sand it will carry and produce a mortar of a given strength.

20.—The degree of final pulverization which the cement receives at the place of manufacture is ascertained by measuring the residue retained on certain sieves. Those known as the No. 100 and No. 200 sieves are recommended for this purpose.

21.—APPARATUS.—The sieves should be circular, about 20 cm. (7.87 ins.) in diameter, 6 cm. (2.36 ins.) high, and provided with a pan 5 cm. (1.97 ins.) deep, and a cover.

22.—The wire cloth should be woven from brass wire having the following diameters:

No. 100, 0.0045 in.; No. 200, 0.0024 in.

23.—This cloth should be mounted on the frames without distortion; the mesh should be regular in spacing and be within the following limits:

No. 100, 96 to 100 meshes to the linear inch.

No. 200, 188 to 200 “ “ “ “ “

24.—Fifty grams (1.76 oz.) or 100 gr. (3.52 oz.) should be used for the test, and dried at a temperature of 100° Cent. (212° Fahr.) prior to sieving.

25.—**METHOD.**—The Committee, after careful investigation, has reached the conclusion that mechanical sieving is not as practicable or efficient as hand work, and, therefore, recommends the following method:

26.—The thoroughly dried and coarsely screened sample is weighed and placed on the No. 200 sieve, which, with pan and cover attached, is held in one hand in a slightly inclined position, and moved forward and backward, at the same time striking the side gently with the palm of the other hand, at the rate of about 200 strokes per minute. The operation is continued until not more than one-tenth of 1 per cent. passes through after one minute of continuous sieving. The residue is weighed, then placed on the No. 100 sieve and the operation repeated. The work may be expedited by placing in the sieve a small quantity of large shot. The results should be reported to the nearest tenth of 1 per cent.

#### **NORMAL CONSISTENCY.**

27.—**SIGNIFICANCE.**—The use of a proper percentage of water in making the pastes\* from which pats, tests of setting and briquettes are made, is exceedingly important, and affects vitally the results obtained.

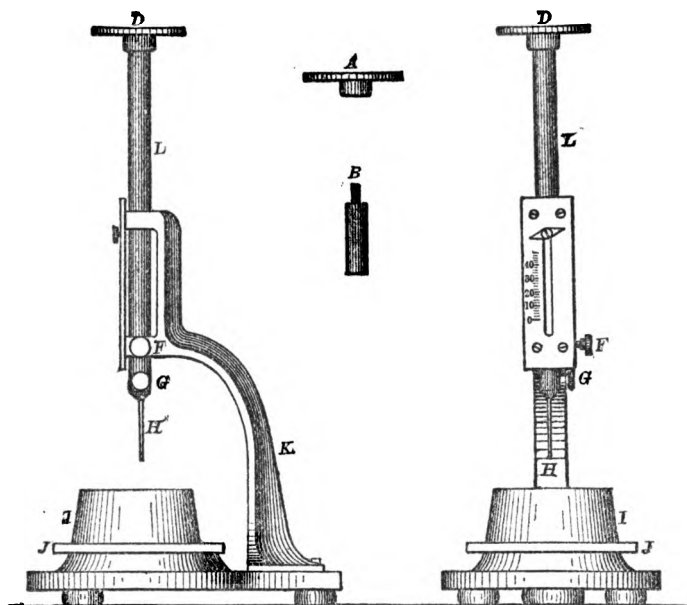
28.—The determination consists in measuring the amount of water required to reduce the cement to a given state of plasticity, or to what is usually designated the normal consistency.

29.—Various methods have been proposed for making this determination, none of which has been found entirely satisfactory. The Committee recommends the following:

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\* The term "paste" is used in this report to designate a mixture of cement and water, and the word "mortar" a mixture of cement, sand and water.

30.—**METHOD. VICAT NEEDLE APPARATUS.**—This consists of a frame (*K*), Fig. 2, bearing a movable rod (*L*), with the cap (*A*) at one end, and at the other the cylinder (*B*), 1 cm. (0.39 in.) in diameter, the cap, rod and cylinder weighing 300 gr. (10.58 oz.). The rod, which can be held in any desired position by a screw (*F*), carries an indicator, which moves over a scale (graduated to centimeters) attached to the frame (*K*). The paste is held by a conical, hard-rubber ring (*I*), 7 cm. (2.76 ins.) in diameter at the base, 4 cm. (1.57 ins.) high, resting on a glass plate (*J*), about 10 cm. (3.94 ins.) square.



VICAT NEEDLE.

FIG. 2.

31.—In making the determination, the same quantity of cement as will be subsequently used for each batch in making the briquettes (but not less than 500 grams) is kneaded into a paste, as described in Paragraph 58, and quickly formed into a ball with the hands, completing the operation by tossing it six times from one hand to the other, maintained 6 ins. apart; the ball is then pressed into the rubber ring, through the larger opening, smoothed off, and placed (on its large end) on a glass plate and the smaller end smoothed off with a trowel; the paste, confined in the ring, resting on the plate, is placed under the rod bearing the cylinder, which is brought in contact with the surface and quickly released.

32.—The paste is of normal consistency when the cylinder pene-

trates to a point in the mass 10 mm. (0.39 in.) below the top of the ring. Great care must be taken to fill the ring exactly to the top.

33.—The trial pastes are made with varying percentages of water until the correct consistency is obtained.

34.—The Committee has recommended, as normal, a paste, the consistency of which is rather wet, because it believes that variations in the amount of compression to which the briquette is subjected in moulding are likely to be less with such a paste.

35.—Having determined in this manner the proper percentage of water required to produce a paste of normal consistency, the proper percentage required for the mortars is obtained from an empirical formula.

36.—The Committee hopes to devise such a formula. The subject proves to be a very difficult one, and, although the Committee has given it much study, it is not yet prepared to make a definite recommendation.

*NOTE. The Committee on Standard Specifications for Cement inserts the following table for temporary use to be replaced by one to be devised by the Committee of the American Society of Civil Engineers.*

PERCENTAGE OF WATER FOR STANDARD SAND MORTARS.

Neat.	One Cement Three Standard Ottawa Sand.	Neat.	One Cement Three Standard Ottawa Sand.	Neat.	One Cement Three Standard Ottawa Sand.
15	8.0	23	9.3	31	10.7
16	8.2	24	9.5	32	10.8
17	8.3	25	9.7	33	11.0
18	8.5	26	9.8	34	11.2
19	8.7	27	10.0	35	11.5
20	8.8	28	10.2	36	11.5
21	9.0	29	10.3	37	11.7
22	9.2	30	10.5	38	11.8

	1 to 1	1 to 2	1 to 3	1 to 4	1 to 5
Cement .....	500	333	250	200	167
Sand .....	500	666	750	800	833

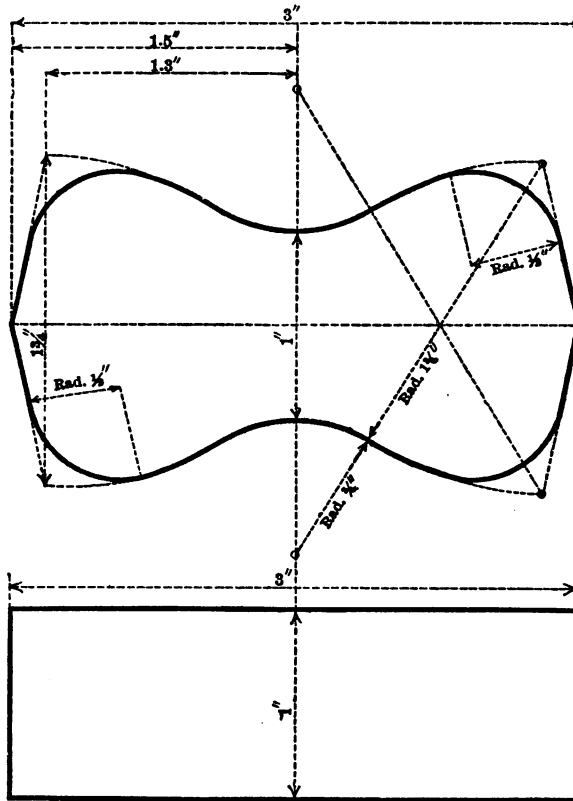
#### TIME OF SETTING.

37.—SIGNIFICANCE.—The object of this test is to determine the time which elapsed from the moment water is added until the paste ceases to be fluid and plastic (called the "initial set"), and also the time required for it to acquire a certain degree of hardness (called the "final"



or "hard set"). The former of these is the more important, since, with the commencement of setting, the process of crystallization or hardening is said to begin. As a disturbance of this process may produce a loss of strength, it is desirable to complete the operation of mixing and moulding or incorporating the mortar into the work before the cement begins to set.

38.—It is usual to measure arbitrarily the beginning and end of the setting by the penetration of weighted wires of given diameters.



DETAILS FOR BRIQUETTE.

FIG. 3.

39.—METHOD.—For this purpose the Vicat Needle, which has already described in Paragraph 30, should be used.

40.—In making the test, a paste of normal consistency is moulded and placed under the rod (*L*); Fig. 2, as described in Paragraph 31; this rod, bearing the cap (*D*) at one end and the needle (*H*), 1 mm.

(0.039 in.) in diameter, at the other, weighing 300 gr. (10.58 oz.). The needle is then carefully brought in contact with the surface of the paste and quickly released.

41.—The setting is said to have commenced when the needle ceases to pass a point 5 mm. (0.20 in.) above the upper surface of the glass plate, and is said to have terminated the moment the needle does not sink visibly into the mass.

42.—The test pieces should be stored in moist air during the test; this is accomplished by placing them on a rack over water contained in a pan and covered with a damp cloth, the cloth to be kept away from them by means of a wire screen; or they may be stored in a moist box or closet.

43.—Care should be taken to keep the needle clean, as the collection of cement on the sides of the needle retards the penetration, while cement on the point reduces the area and tends to increase the penetration.

44.—The determination of the time of setting is only approximate, being materially affected by the temperature of the mixing water, the temperature and humidity of the air during the test, the percentage of water used, and the amount of moulding the paste receives.

#### STANDARD SAND.

45.—The Committee recognizes the grave objections to the standard quartz now generally used, especially on account of its high percentage of voids, the difficulty of compacting in the moulds, and its lack of uniformity; it has spent much time in investigating the various natural sands which appeared to be available and suitable for use.

46.—For the present, the Committee recommends the natural sand from Ottawa, Ill., screened to pass a sieve having 20 meshes per linear inch and retained on a sieve having 30 meshes per linear inch; the wires to have diameters of 0.0165 and 0.0112 in., respectively, *i. e.*, half the width of the opening in each case. Sand having passed the No. 20 sieve shall be considered standard when not more than one per cent. passes a No. 30 sieve after one minute continuous sifting of a 500-gram sample.

47.—The Sandusky Portland Cement Company, of Sandusky, Ohio, has agreed to undertake the preparation of this sand, and to furnish it at a price only sufficient to cover the actual cost of preparation.

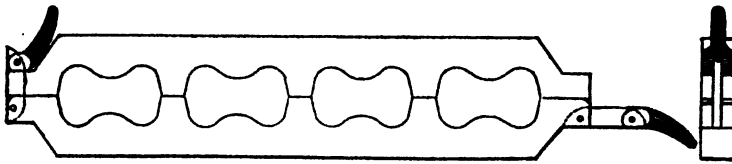
#### FORM OF BRIQUETTE.

48.—While the form of the briquette recommended by a former Committee of the Society is not wholly satisfactory, this Committee

is not prepared to suggest any change, other than rounding off the corners by curves of  $\frac{1}{2}$ -in. radius, Fig. 3.

### MOULDS.

49.—The moulds should be made of brass, bronze or some equally non-corrodible material, having sufficient metal in the sides to prevent spreading during moulding.



DETAILS FOR GANG MOULD.

FIG. 4.

50.—Gang moulds, which permit moulding a number of briquettes at one time, are preferred by many to single moulds; since the greater quantity of mortar that can be mixed tends to produce greater uniformity in the results. The type shown in Fig. 4 is recommended.

51.—The moulds should be wiped with an oily cloth before using.

### MIXING.

52.—All proportions should be stated by weight; the quantity of water to be used should be stated as a percentage of the dry material.

53.—The metric system is recommended because of the convenient relation of the gram and the cubic centimeter.

54.—The temperature of the room and the mixing water should be as near 21 degrees Cent. (70 degrees Fahr.) as it is practicable to maintain it.

55.—The sand and cement should be thoroughly mixed dry. The mixing should be done on some non-absorbing surface, preferably plate glass. If the mixing must be done on an absorbing surface it should be thoroughly dampened prior to use.

56.—The quantity of material to be mixed at one time depends on the number of test pieces to be made; about 1000 gr. (35.28 oz.) makes a convenient quantity to mix, especially by hand methods.

57.—The Committee, after investigation of the various mechanical mixing machines, has decided not to recommend any machine that has thus far been devised, for the following reasons:

(1) The tendency of most cement is to "ball up" in the machine,

thereby preventing the working of it into a homogeneous paste; (2) there are no means of ascertaining when the mixing is complete without stopping the machine, and (3) the difficulty of keeping the machine clean.

58.—**METHOD.**—The material is weighed and placed on the mixing table, and a crater formed in the center, into which the proper percentage of clean water is poured; the material on the outer edge is turned into the crater by the aid of a trowel. As soon as the water has been absorbed, which should not require more than one minute, the operation is completed by vigorously kneading with the hands for an additional  $1\frac{1}{2}$  minutes, the process being similar to that used in kneading dough. A sand-glass affords a convenient guide for the time of kneading. During the operation of mixing the hands should be protected by gloves, preferably of rubber.

#### MOULDING.

59.—Having worked the paste or mortar to the proper consistency, it is at once placed in the moulds by hand.

60.—The Committee has been unable to secure satisfactory results with the present moulding machines; the operation of machine moulding is very slow, and the present types permit of moulding but one briquette at a time, and are not practicable with the pastes or mortars herein recommended.

61.—**Method.**—The moulds should be filled at once, the material pressed in firmly with the fingers and smoothed off with a trowel without ramming; the material should be heaped up on the upper surface of the mould, and, in smoothing off, the trowel should be drawn over the mould in such a manner as to exert a moderate pressure on the excess material. The mould should be turned over and the operation repeated.

62.—A check upon the uniformity of the mixing and moulding is afforded by weighing the briquettes just prior to immersion, or upon removal from the moist closet. Briquettes which vary in weight more than 3 per cent. from the average should not be tested.

#### STORAGE OF THE TEST PIECES.

63.—During the first 24 hours after moulding the test pieces should be kept in moist air to prevent them from drying out.

64.—A moist closet or chamber is so easily devised that the use of the damp cloth should be abandoned if possible. Covering the test pieces with a damp cloth is objectionable, as commonly used, because

the cloth may dry out unequally, and, in consequence, the test pieces are not all maintained under the same condition. Where a moist

closet is not available, a cloth may be used and kept uniformly wet by immersing the ends in water. It should be kept from direct contact with the test pieces by means of a wire screen or some similar arrangement.

65.—A moist closet consists of a soapstone or slate box, or a metal-lined wooden box—the metal lining being covered with felt and this felt kept wet. The bottom of the box is so constructed as to hold water, and the sides are provided with cleats for holding glass shelves on which to place the briquettes. Care should be taken to keep the air in the closet uniformly moist.

66.—After 24 hours in moist air the test pieces for longer periods of time should be immersed in water maintained as near  $21^{\circ}$  Cent. ( $70^{\circ}$  Fahr.) as practicable; they may be stored in tanks or pans, which should be of non-corrodible material.

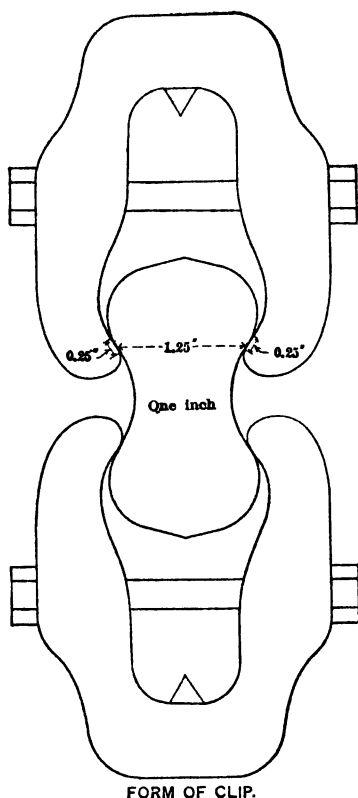


FIG. 5.

#### TENSILE STRENGTH.

67.—The tests may be made on any standard machine. A solid metal clip, as shown in Fig. 5, is recommended. This clip is to be used without cushioning at the points of contact with the test specimen. The bearing at each point of contact should be  $\frac{1}{4}$  in. wide, and the distance between the center of contact on the same clip should be  $1\frac{1}{4}$  ins.

68.—Test pieces should be broken as soon as they are removed from the water. Care should be observed in centering the briquettes in the testing machine, as cross-strains, produced by improper centering, tend to lower the breaking strength. The load should not be applied too suddenly, as it may produce vibration, the shock from which often breaks the briquette before the ultimate strength is reached. Care must be taken that the clips and the sides of the

briquette be clean and free from grains of sand or dirt, which would prevent a good bearing. The load should be applied at the rate of 600 lbs. per minute. The average of the briquettes of each sample tested should be taken as the test, excluding any results which are manifestly faulty.

#### CONSTANCY OF VOLUME.

69.—SIGNIFICANCE.—The object is to develop those qualities which tend to destroy the strength and durability of a cement. As it is highly essential to determine such qualities at once, tests of this character are for the most part made in a very short time, and are known, therefore, as accelerated tests. Failure is revealed by cracking, checking, swelling or disintegration, or all of these phenomena. A cement which remains perfectly sound is said to be of constant volume.

70.—METHODS.—Tests for constancy of volume are divided into two classes: (1) normal tests, or those made in either air or water maintained at about 21° Cent. (70° Fehr.), and (2) accelerated tests, or those made in air, steam or water at a temperature of 45° Cent. (115° Fahr.) and upward. The test pieces should be allowed to remain 24 hours in moist air before immersion in water or steam, or preservation in air.

71.—For these tests, pats, about  $7\frac{1}{2}$  cm. (2.95 ins.) in diameter,  $1\frac{1}{4}$  cm. (0.49 in.) thick at the center, and tapering to a thin edge, should be made, upon a clean glass plate [about 10 cm. (3.94 ins.) square], from cement paste of normal consistency.

72.—NORMAL TEST.—A pat is immersed in water maintained as near 21° Cent. (70° Fahr.) as possible for 28 days, and observed at intervals. A similar pat is maintained in air at ordinary temperature and observed at intervals.

73.—ACCELERATED TEST.—A pat is exposed in any convenient way in an atmosphere of steam, above boiling water, in a loosely closed vessel, for 3 hours.

74.—To pass these tests satisfactorily, the pats should remain firm and hard, and show no signs of cracking, distortion or disintegration.

75.—Should the pat leave the plate, distortion may be detected best with a straight-edge applied to the surface which was in contact with the plate.

76.—In the present state of our knowledge it cannot be said that cement should necessarily be condemned simply for failure to pass the

accelerated tests; nor can a cement be considered entirely satisfactory, simply because it has passed these tests.

Submitted on behalf of the Committee,

GEORGE S. WEBSTER,  
*Chairman.*

RICHARD L. HUMPHREY,  
*Secretary.*

*Committee.*

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Richard L. Humphrey,  
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F. H. Lewis.



## *New York Section* *Society for Chemical Industry*

METHOD SUGGESTED FOR THE ANALYSIS OF LIMESTONES, RAW MIXTURES AND PORTLAND CEMENTS BY THE COMMITTEE  
ON UNIFORMITY IN TECHNICAL ANALYSIS WITH  
THE ADVICE OF W. F. HILLEBRAND.

---

### **SOLUTION.**

One-half gram of the finely-powdered substance is to be weighed out and, if a limestone or unburned mixture, strongly ignited in a covered platinum crucible over a strong blast for 15 minutes, or longer if the blast is not powerful enough to effect complete conversion to a cement in this time. It is then transferred to an evaporating dish, preferably of platinum for the sake of celerity in evaporation, moistened with enough water to prevent lumping, and 5 to 10 c. c. of strong HCl added and digested with the aid of gentle heat and agitation until solution is complete. Solution may be aided by light pressure with the flattened end of a glas rod.\* The solution is then evaporated to dryness, as far as this may be possible on the bath.

### **SILICA ( $\text{SiO}_2$ ).**

The residue without further heating is treated at first with 5 to 10 c. c. of strong HCl, which is then diluted to half strength or less, or upon the residue may be poured at once a larger volume of acid of half strength. The dish is then covered and digestion allowed to go on for 10 minutes on the bath, after which the solution is filtered and the separated silica washed thoroughly with water. The filtrate is again evaporated to dryness, the residue without further heating, taken up with acid and water and the small amount of silica it contains separated on another filter paper. The papers containing the residue are transferred wet to a weighed platinum crucible, dried, ignited, first over a Bunsen burner until the carbon of the filter is completely consumed, and finally over the blast for 15 minutes and checked by a further blasting for 10 minutes or to constant weight.

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\* If anything remains undecomposed it should be separated, fused with a little  $\text{Na}_2\text{CO}_3$ , dissolved and added to the original solution. Of course a small amount of separated non-gelatinous silica is not to be mistaken for undecomposed matter.

The silica, if great accuracy is desired, is treated in the crucible with about 10 c. c. of  $\text{HFl}$  and four drops of  $\text{H}_2\text{SO}_4$  and evaporated over a low flame to complete dryness. The small residue is finally blasted, for a minute or two, cooled and weighed. The difference between this weight and the weight previously obtained gives the amount of silica.\*

#### ALUMINA AND IRON ( $\text{Al}_2\text{O}_3$ AND $\text{Fe}_2\text{O}_3$ ).

The filtrate, about 250 c. c., from the second evaporation for  $\text{SiO}_2$ , is made alkaline with  $\text{NH}_4\text{OH}$  after adding  $\text{HCl}$ , if need be, to insure a total of 10 to 15 c. c. strong acid, and boiled to expel excess of  $\text{NH}_3$ , or until there is but a faint odor of it, and the precipitate iron and aluminum hydrates, after settling, are washed once by decantation and slightly on the filter. Setting aside the filtrate, the precipitate is dissolved in hot dilute  $\text{HCl}$ , the solution passing into the beaker in which the precipitation was made. The aluminum and iron are then reprecipitated by  $\text{NH}_4\text{OH}$ , boiled and the second precipitate collected and washed on the same filter used in the first instance. The filter paper, with the precipitate, is then placed in a weighed platinum crucible, the paper burned off and the precipitate ignited and finally blasted 5 minutes, with care to prevent reduction, cooled and weighed as  $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ .†

#### IRON ( $\text{Fe}_2\text{O}_3$ ).

The combined iron and aluminum oxides are fused in a platinum crucible at a very low temperature with about 3 to 4 grams of  $\text{KHSO}_4$ , or, better,  $\text{NaHSO}_4$ , the melt taken up with so much dilute  $\text{H}_2\text{SO}_4$  that there shall be no less than 5 grams absolute acid and enough water to effect solution on heating. The solution is then evaporated and eventually heated till acid fumes come off copiously. After cooling and redissolving in water the small amount of silica is filtered out, weighed and corrected by  $\text{HFl}$  and  $\text{H}_2\text{SO}_4$ .‡ The filtrate is reduced by zinc, or preferably by hydrogen sulphide, boiling out the excess of the latter afterwards while passing  $\text{CO}_2$  through the flask, and titrated with permanganate.§ The strength of the permanganate solution should not be greater than .0040 gr.  $\text{Fe}_2\text{O}_3$  per c. c.

\* For ordinary control in the plant laboratory this correction may, perhaps, be neglected; the double evaporation never.

† This precipitate contains  $\text{TiO}_2$ ,  $\text{P}_2\text{O}_5$ ,  $\text{Mn}_2\text{O}_4$ .

‡ This correction of  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  for silica should not be made when the  $\text{HFl}$  correction of the main silica has been omitted, unless that silica was obtained by only one evaporation and filtration. After two evaporations and filtrations 1 to 2 mg. of  $\text{SiO}$  are still to be found with the  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ .

§ In this way only is the influence of titanium to be avoided and a correct result obtained for iron.

**LIME (CaO)**

To the combined filtrate from the  $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  precipitate a few drops of  $\text{NH}_4\text{OH}$  are added, and the solution brought to boiling. To the boiling solution 20 c. c. of a saturated solution of ammonium oxalate are added, and the boiling continued until the precipitated  $\text{CaC}_2\text{O}_4$  assumes a well-defined granular form. It is then allowed to stand for 20 minutes, or until the precipitate has settled, and then filtered and washed. The precipitate and filter are placed wet in a platinum crucible, and the paper burned off over a small flame of a Bunsen burner. It is then ignited, redissolved in  $\text{HCl}$ , and the solution made up to 100 c. c. with water. Ammonia is added in slight excess, and the liquid is boiled. If a small amount of  $\text{Al}_2\text{O}_3$  separates this is filtered out, weighed, and the amount added to that found in the first determination, when greater accuracy is desired. The lime is then reprecipitated by ammonium oxalate, allowed to stand until settled, filtered, and washed,\* weighed as oxide by ignition and blasting in a covered crucible to constant weight, or determined with dilute standard permanganate.†

**MAGNESIA (MgO).**

The combined filtrates from the calcium precipitates are acidified with  $\text{HCl}$  and concentrated on the steam bath to about 150 c. c., 10 c. c. of saturated solution of  $\text{Na}(\text{NH}_4)\text{HPO}_4$  are added, and the solution boiled for several minutes. It is then removed from the flame and cooled by placing the beaker in ice water. After cooling,  $\text{NH}_4\text{OH}$  is added drop by drop with constant stirring until the crystalline ammonium-magnesium ortho-phosphate begins to form, and then in moderate excess, the stirring being continued for several minutes. It is then set aside for several hours in a cool atmosphere and filtered. The precipitate is redissolved in hot dilute  $\text{HCl}$ , the solution made up to about 100 c. c., 1 c. c. of a saturated solution of  $\text{Na}(\text{NH}_4)\text{HPO}_4$  added, and ammonia drop by drop, with constant stirring until the precipitate is again formed as described and the ammonia is in moderate excess. It is then allowed to stand for about 2 hours, when it is filtered on a paper or a Gooch crucible, ignited, cooled and weighed as  $\text{Mg}_2\text{P}_2\text{O}_7$ .

**ALKALIES ( $\text{K}_2\text{O}$  AND  $\text{Na}_2\text{O}$ ).**

For the determination of the alkalies, the well-known method of Prof. J. Lawrence Smith is to be followed, either with or without the addition of  $\text{CaCO}_3$  with  $\text{NH}_4\text{Cl}$ .

\* The volume of wash-water should not be too large; vide Hildebrand.

† The accuracy of this method admits of criticism, but its convenience and rapidity demand its insertion.

**ANHYDROUS SULPHURIC ACID ( $\text{SO}_3$ ).**

One gram of the substance is dissolved in 15 c. c. of  $\text{HCl}$ , filtered and residue washed thoroughly.\*

The solution is made up to 250 c. c. in a beaker and boiled. To the boiling solution 10 c. c. of a saturated solution of  $\text{BaCl}_2$  is added slowly drop by drop from a pipette and the boiling continued until the precipitate is well formed, or digestion on the steam bath may be substituted for the boiling. It is then set aside over night, or for a few hours, filtered, ignited and weighed as  $\text{BaSO}_4$ .

**TOTAL SULPHUR.**

One gram of the material is weighed out in a large platinum crucible and fused with  $\text{Na}_2\text{CO}_3$  and a little  $\text{KNO}_3$  being careful to avoid contamination from sulphur in the gases from source of heat. This may be done by fitting the crucible in a hole in an asbestos board. The melt is treated in the crucible with boiling water and the liquid poured into a tall narrow beaker and more hot water added until the mass is disintegrated. The solution is then filtered. The filtrate contained in a No. 4 beaker is to be acidulated with  $\text{HCl}$  and made up to 250 c. c. with distilled water, boiled, the sulphur precipitated as  $\text{BaSO}_4$  and allowed to stand over night or for a few hours.

**LOSS ON IGNITION.**

Half a gram of cement is to be weighed out in a platinum crucible, placed in a hole in an asbestos board so that about  $\frac{3}{8}$  of the crucible projects below, and blasted 15 minutes, preferably with an inclined flame. The loss by weight, which is checked by a second blasting of 5 minutes, is the loss on ignition.

May, 1903: Recent investigations have shown that large errors in results are often due to the use of impure distilled water and reagents. The analyst should, therefore, test his distilled water by evaporation and his reagents by appropriate tests before proceeding with his work.

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\* Evaporation to dryness is unnecessary, unless gelatinous silica should have separated and should never be performed on a bath heated by gas; vide Hillebrand.

# American Society for Testing Materials

## *Committee on Standard Specifications for Cement*

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**H. G. KELLY, Vice-President American Railway Engineering and Maintenance of Way Association, Minneapolis, Minn.**

## *Report of Committee*

ADOPTED BY THE SOCIETY, NOVEMBER 14TH, 1904.

---

### **GENERAL OBSERVATIONS.**

1. These remarks have been prepared with a view of pointing out the pertinent features of the various requirements and the precautions to be observed in the interpretation of the results of the tests.

2. The Committee would suggest that the acceptance or rejection under these specifications be based on tests made by an experienced person having the proper means for making the tests.

### **SPECIFIC GRAVITY.**

Specific gravity is useful in detecting adulteration or underburning. The results of tests of specific gravity are not necessarily conclusive as an indication of the quality of a cement, but when in combination with the results of other tests may afford valuable indications.

### **FINENESS.**

4. The sieves should be kept thoroughly dry.

### **TIME OF SETTING.**

5. Great care should be exercised to maintain the test pieces under as uniform conditions as possible. A sudden change or wide range of temperature in the room in which the tests are made, a very dry or humid atmosphere, and other irregularities vitally affect the rate of setting.

### **TENSILE STRENGTH.**

6. Each consumer must fix the minimum requirements for tensile strength to suit his own conditions. They shall, however, be within the limits stated.

### **CONSTANCY OF VOLUME.**

7. The tests for constancy of volume are divided into two classes, the first normal, the second accelerated. The latter should be regarded as a precautionary test only, and not infallible. So many conditions enter into the making and interpreting of it that it should be used with extreme care.

8. In making the pats the greatest care should be exercised to avoid initial strains due to molding or to too rapid drying-out during the first twenty-four hours. The pats should be preserved under the most uniform conditions possible, and rapid changes of temperature should be avoided.

9. The failure to meet the requirements of the accelerated tests need not be sufficient cause for rejection. The cement may, however, be held for twenty-eight days, and a retest made at the end of that period. Failure to meet the requirements at this time should be considered sufficient cause for rejection, although in the present state of our knowledge it cannot be said that such failure necessarily indicates unsoundness, nor can the cement be considered entirely satisfactory simply because it passes the tests.

### GENERAL CONDITIONS.

- 1        1. All cement shall be inspected.
- 2        2. Cement may be inspected either at the place of manu-  
3        facture or on the work.
- 4        3. In order to allow ample time for inspecting and testing,  
5        the cement should be stored in a suitable weather-tight building  
6        having the floor properly blocked or raised from the ground.
- 7        4. The cement shall be stored in such a manner as to  
8        permit easy access for proper inspection and identification of  
9        each shipment.
- 10       5. Every facility shall be provided by the contractor and a  
11       period of at least twelve days allowed for the inspection and  
12       necessary tests.
- 13       6. Cement shall be delivered in suitable packages with the  
14       brand and name of manufacturer plainly marked thereon.
- 15       7. A bag of cement shall contain 94 pounds of cement net.  
16       Each barrel of Portland cement shall contain 4 bags, and each  
17       barrel of natural cement shall contain 3 bags of the above net  
18       weight.
- 19       8. Cement failing to meet the seven-day requirements may  
20       be held awaiting the results of the twenty-eight day tests before  
21       rejection.
- 22       9. All tests shall be made in accordance with the methods  
23       proposed by the Committee on Uniform Tests of Cement of the  
24       American Society of Civil Engineers, presented to the Society  
25       January 21, 1903, and amended January 20, 1904, with all  
26       subsequent amendments thereto.
- 27       10. The acceptance or rejection shall be based on the  
28       following requirements:



**NATURAL CEMENT.**

29 11. *Definition.* This term shall be applied to the finely  
 30 pulverized product resulting from the calcination of an argil-  
 31 laceous limestone at a temperature only sufficient to drive off  
 32 the carbonic acid gas.

**SPECIFIC GRAVITY.**

33 12. The specific gravity of the cement thoroughly dried at  
 34 100° C., shall be not less than 2.8.

**FINENESS.**

35 13. It shall leave by weight a residue of not more than  
 36 10% on the No. 100, and 30% on the No. 200 sieve.

**TIME OF SETTING.**

37 14. It shall develop initial set in not less than ten minutes,  
 38 and hard set in not less than thirty minutes, nor more than  
 39 three hours.

**TENSILE STRENGTH.**

40 15. The minimum requirements for tensile strength for  
 41 briquettes one inch square in cross section shall be within the  
 42 following limits, and shall show no retrogression in strength  
 43 within the periods specified:\*

44 Age.	Neat Cement.	Strength.
45 24 hours in moist air .....		50-100 lbs.
46 7 days (1 day in moist air, 6 days in water) ..		100-200 "
47 28 days (1 day in moist air, 27 days in water) ..		200-300 "
48	<i>One Part Cement, Three Parts Standard Sand.</i>	
49 7 days (1 day in moist air, 6 days in water) ..		25- 75 "
50 28 days (1 day in moist air, 27 days in water) ..		75-150 "

**CONSTANCY OF VOLUME.**

51 16. Pats of neat cement about three inches in diameter,  
 52 one-half inch thick at centre, tapering to a thin edge, shall be  
 53 kept in moist air for a period of twenty-four hours.  
 54 (a) A pat is then kept in air at normal temperature.  
 55 (b) Another is kept in water maintained as near 70° F. as  
 56 practicable.

---

\* For example the minimum requirement for the twenty-four hour neat cement test should be some specified value within the limits of 50 and 100 pounds, and so on for each period stated.

17. These pats are observed at intervals for at least 28 days, and, to satisfactorily pass the tests, should remain firm and hard and show no signs of distortion, checking, cracking or disintegrating.

### PORTLAND CEMENT.

18. *Definition.* This term is applied to the finely pulverized product resulting from the calcination to incipient fusion of an intimate mixture of properly proportioned argillaceous and calcareous materials, and to which no addition greater than 3% has been made subsequent to calcination.

### SPECIFIC GRAVITY.

19. The specific gravity of the cement, thoroughly dried at 100° C., shall be not less than 3.10.

### FINENESS.

20. It shall leave by weight a residue of not more than 8% on the No. 100, and not more than 25% on the No. 200 sieve.

### TIME OF SETTING.

21. It shall develop initial set in not less than thirty minutes, but must develop hard set in not less than one hour, nor more than ten hours.

### TENSILE STRENGTH.

22. The minimum requirements for tensile strength for briquettes one inch square in section shall be within the following limits, and shall show no retrogression in strength within the periods specified:\*

Age.	Neat Cement.	Strength.
24 hours in moist air	.....	150-200 lbs.
7 days (1 day in moist air, 6 days in water)	..	450-550 "
28 days (1 day in moist air, 27 days in water)	..	550-650 "
<i>One Part Cement, Three Parts Sand.</i>		
7 days (1 day in moist air, 6 days in water)	..	150-200 lbs.
28 days (1 day in moist air, 27 days in water)	..	200-300 "

\* For example the minimum requirement for the twenty-four hour neat cement test should be some specified value within the limits of 150 and 200 pounds, and so on for each period stated.

**CONSTANCY OF VOLUME.**

84 23. Pats of neat cement about three inches in diameter,  
85 one-half inch thick at the centre, and tapering to a thin edge,  
86 shall be kept in moist air for a period of twenty-four hours.

87 (a) A pat is then kept in air at normal temperature and  
88 observed at intervals for at least 28 days.

89 (b) Another pat is kept in water maintained as near 70°  
90 F. as practicable, and observed at intervals for at least 28 days.

91 (c) A third pat is exposed in any convenient way in an  
92 atmosphere of steam, above boiling water, in a loosely closed  
93 vessel for five hours.

94 24. These pats, to satisfactorily pass the requirements,  
95 shall remain firm and hard and show no signs of distortion,  
96 checking, cracking or disintegrating.

**SULPHURIC ACID AND MAGNESIA.**

97 25. The cement shall not contain more than 1.75% of  
98 anhydrous sulphuric acid ( $\text{SO}_3$ ), nor more than 4% of mag-  
99 nesia ( $\text{MgO}$ ).

Submitted on behalf of the committee.

GEORGE F. SWAIN, *Chairman.*

GEORGE S. WEBSTER, *Vice-Chairman.*

RICHARD L. HUMPHREY, *Secretary.*

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